

MEMORANDUM

TO: Terry Taylor
Anderson, Mulholland and Associates

DATE: February 19, 2012

FROM: R. Infante

FILE: JA98695

RE: Data Validation
BMSMC, Building 5 Area, PR
SM04.00.06

Accutest Job Number: JA98695

SUMMARY

Full validation was performed on the data for (2) soil samples for selected volatile organic compounds using EPA method SW-846 8260B and for alcohols (methanol and isopropyl alcohol) by EPA method SW-846 8015 (DAI). The samples were collected at the BMSMC Building 5 Area in Humacao, PR on February 2, 2012 and submitted to Accutest Laboratories that analyzed and reported the results under delivery group (SDG) JA98695.

The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: *USEPA Region 2, SOP HW-24, Standard Operating Procedure for the Validation of Organic Data Acquired using SW-846 Method 8260B (August 2009 Revision 2)* and the *USEPA National Functional Guidelines for Low/Medium Concentration Organic Data Review (SOW SOM01.2 SOP HW-33, August 2009 - Revision 2)*, the *USEPA National Functional Guidelines for Organic Data Review for Low Concentration Water (SOP HW-13, August 2009-Revision 3)*, (noted herein as the "primary guidance document"). Also the QC criteria from *"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW-846 (Final Update III, December 1996)"*. The guidelines were modified to accommodate the non-CLP methodology. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

In general the data is valid as reported and may be used for decision making purposes. The data results are acceptable for use. Some of the results were qualified.

SAMPLES

The samples included in the review are listed below

FIELD SAMPLE ID	LABORATORY ID	ANALYSIS
I-7A	JA98695-1	VOCs, ALCOHOLS
I-1R3A	JA98695-2	VOCs, ALCOHOLS

REVIEW ELEMENTS

Sample data were reviewed for the following parameters, where applicable to the method

- Agreement of analysis conducted with chain of custody (COC) form
- Holding time and sample preservation
- Gas chromatography/mass spectrometry (GC/MS) tunes
- Initial and continuing calibrations
- Method blanks/trip blanks/field blank
- Surrogate spike recovery
- Matrix spike/matrix spike duplicate (MS/MSD) results
- Internal standard performance
- Field duplicate results
- Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results
- Quantitation limits and sample results

DISCUSSION

Agreement of Analysis Conducted with COC Request

Sample reports corresponded to the analytical request designated on the chain-of-custody form. However, the sample identified as A-1R3A was reported as I-1R3A.

Holding Times and Sample Preservation

The cooler temperatures were within the QC acceptance criteria of $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

Sample preservation was acceptable.

Samples analyzed within method recommended holding time.

GC/MS Tunes

The frequency and abundance of bromofluorobenzene (BFB) tunes were within the QC acceptance criteria. All samples were analyzed within the tuning criteria associated with the method.

Initial and Continuing Calibrations

VOCs

The percent relative standard deviations (%RSDs) and response factors (RFs) of all target analytes were within the QC acceptance criteria in the initial calibration. Correlation coefficients (r^2) of target analytes were within the QC acceptance criteria. Ongoing accuracy of the instrument was determined by the analysis of a continuing calibration standard. All initial and continuing calibrations met the acceptance criteria.

Alcohols

The percent relative standard deviations (%RSDs) and response factors (RFs) of all target analytes were within the QC acceptance criteria in the initial calibration. Correlation coefficients (r^2) of target analytes were within the QC acceptance criteria. Ongoing accuracy of the instrument was determined by the analysis of a continuing calibration standard. All initial and continuing calibrations met the acceptance criteria.

Method Blank/Trip Blank/Field Blank

Target analytes were not detected in laboratory method blanks for VOCs and alcohols.

No trip/field/equipment blanks associated with this data set (VOCs and alcohols).

Surrogate Spike Recovery

The surrogate recoveries were within the laboratory QC acceptance limits in all samples analyzed for VOCs.

The surrogate recoveries were within the laboratory QC acceptance limits in all samples analyzed for alcohols except for the following:

- % recovery for Hexanol in sample JA98695-1 outside the control limits; 42, control limits: 58 – 137 %. No action taken; recoveries within control limits in signal #2.

MS/MSD

VOCs

Matrix spike was performed on samples JA98960-6MS/6MSD and JA98956-3MS/-3MSD were within laboratory control except for the following:

JA98956-3MS/-3MSD

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
MS	ACETONE	236	12	- 189	Qualify_result_(J)_

Results qualified (J) for acetone in sample JA98695-2.

Alcohols

Matrix spike was performed on samples JA98540-8MS/8MSD and JA98540-2MS/-2MSD. Recoveries and RPD for the MS/MSD were within laboratory control limits except for the followings:

JA98540-2MS/-2MSD

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
MS	METHANOL	193	39	- 160	No_action__
MSD		195			

No action taken; methanol was not detected in the samples.

Internal Standard Performance

VOCs

Samples were spiked with the method specified internal standard. Internal standard performance met the QC acceptance criteria in all sample analyses.

Laboratory/Field Duplicate Results

Laboratory duplicate associated with data package were samples JA98956-8/-8DUP (VOCs); no field/laboratory duplicates analyzed for alcohols. VOCs RPD results were within laboratory and generally acceptable control limits. MS/MSD results were used to assess precision for alcohols, RPD results were within laboratory and generally acceptable control limits.

LCS/LCSD Results

VOCs

The laboratory analyzed one LCS (blank spike) associated with each matrix from this data set. The % recoveries of all spiked analytes were within the laboratory QC acceptance limits.

Alcohols

The laboratory analyzed one LCS (blank spike) associated with each matrix from this data set. The % recoveries of all spiked analytes were within the laboratory QC acceptance limits.

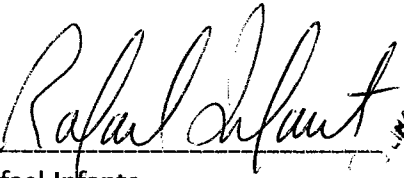
Quantitation Limits and Sample Results


Dilutions were not required with this data set.

Calculations were spot checked.

Certification

The following samples JA98695-1 and JA98695-2 were analyzed following standard procedures accepted by regulatory agencies. The quality control requirements met the methods criteria except in the occasions described in this document. The results are valid.


Rafael Infante
Chemist License 1888



Report of Analysis

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Client Sample ID:	I-7A	Date Sampled:	02/02/12
Lab Sample ID:	JA98695-1	Date Received:	02/04/12
Matrix:	SO - Soil	Percent Solids:	82.3
Method:	SW846 8260B SW846 5035		
Project:	BMSMC, Building 5 Area, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	X123648.D	1	02/11/12	TYG	02/04/12 14:00	n/a	VX5283
Run #2							

Run #	Initial Weight
Run #1	5.5 g
Run #2	

VOA Special List

CAS No.	Compound	Result	RL	MDL	Units	Q
67-64-1	Acetone	ND	11	7.3	ug/kg	
71-43-2	Benzene	ND	1.1	0.15	ug/kg	
100-41-4	Ethylbenzene	ND	1.1	0.16	ug/kg	
108-10-1	4-Methyl-2-pentanone(MIBK)	ND	5.5	2.9	ug/kg	
108-88-3	Toluene	ND	1.1	0.42	ug/kg	
1330-20-7	Xylene (total)	ND	1.1	0.20	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
1868-53-7	Dibromofluoromethane	106%		67-131%
17060-07-0	1,2-Dichloroethane-D4	95%		66-130%
2037-26-5	Toluene-D8	114%		76-125%
460-00-4	4-Bromofluorobenzene	99%		53-142%



ND = Not detected MDL - Method Detection Limit
 RL = Reporting Limit
 E = Indicates value exceeds calibration range

J = Indicates an estimated value
 B = Indicates analyte found in associated method blank
 N = Indicates presumptive evidence of a compound

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Report of Analysis

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Client Sample ID:	I-7A	Date Sampled:	02/02/12
Lab Sample ID:	JA98695-1	Date Received:	02/04/12
Matrix:	SO - Soil	Percent Solids:	82.3
Method:	SW846-8015 (DAI)		
Project:	BMSMC, Building 5 Area, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	GH86344.D	1	02/09/12	XPL	n/a	n/a	GGH3960
Run #2							

Run #	Initial Weight
Run #1	5.0 g
Run #2	

CAS No.	Compound	Result	RL	MDL	Units	Q
67-63-0	Isopropyl Alcohol	ND	120	46	ug/kg	
67-56-1	Methanol	ND	240	62	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
111-27-3	Hexanol	42% ^a		58-137%
111-27-3	Hexanol	61%		58-137%

(a) Outside in house QC limits. Data reported from the second channel.



ND = Not detected MDL - Method Detection Limit
RL = Reporting Limit
E = Indicates value exceeds calibration range

J = Indicates an estimated value
B = Indicates analyte found in associated method blank
N = Indicates presumptive evidence of a compound

Report of Analysis

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Client Sample ID:	I-1R3A	Date Sampled:	02/02/12
Lab Sample ID:	JA98695-2	Date Received:	02/04/12
Matrix:	SO - Soil	Percent Solids:	80.4
Method:	SW846 8260B SW846 5035		
Project:	BMSMC, Building 5 Area, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	V124257A.D	1	02/13/12	CL	02/04/12 14:00	n/a	VV5348
Run #2							

Run #	Initial Weight
Run #1	5.8 g
Run #2	

VOA Special List

CAS No.	Compound	Result	RL	MDL	Units	Q
67-64-1	Acetone	29.6 J	11	7.1	ug/kg	
71-43-2	Benzene	ND	1.1	0.14	ug/kg	
100-41-4	Ethylbenzene	12.1	1.1	0.16	ug/kg	
108-10-1	4-Methyl-2-pentanone(MIBK)	ND	5.4	2.8	ug/kg	
108-88-3	Toluene	ND	1.1	0.41	ug/kg	
1330-20-7	Xylene (total)	34.6	1.1	0.20	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
1868-53-7	Dibromofluoromethane	105%		67-131%
17060-07-0	1,2-Dichloroethane-D4	99%		66-130%
2037-26-5	Toluene-D8	110%		76-125%
460-00-4	4-Bromofluorobenzene	99%		53-142%



ND = Not detected MDL - Method Detection Limit
 RL = Reporting Limit
 E = Indicates value exceeds calibration range

J = Indicates an estimated value
 B = Indicates analyte found in associated method blank
 N = Indicates presumptive evidence of a compound

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Report of Analysis

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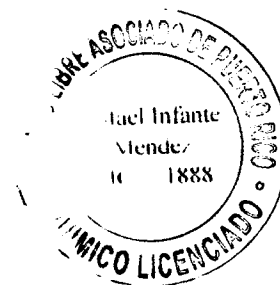
Client Sample ID:	I-1R3A	Date Sampled:	02/02/12
Lab Sample ID:	JA98695-2	Date Received:	02/04/12
Matrix:	SO - Soil	Percent Solids:	80.4
Method:	SW846-8015 (DAI)		
Project:	BMSMC, Building 5 Area, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	GH86291.D	1	02/07/12	XPL	n/a	n/a	GGH3957
Run #2							

Run #	Initial Weight
Run #1	5.0 g
Run #2	

CAS No.	Compound	Result	RL	MDL	Units	Q
67-63-0	Isopropyl Alcohol	ND	120	47	ug/kg	
67-56-1	Methanol	ND	250	64	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
111-27-3	Hexanol	90%		58-137%
111-27-3	Hexanol	112%		58-137%



ND = Not detected MDL - Method Detection Limit
RL = Reporting Limit
E = Indicates value exceeds calibration range

J = Indicates an estimated value
B = Indicates analyte found in associated method blank
N = Indicates presumptive evidence of a compound



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Bottle Order Control ●

Accusant Job # TA98 695

[illegible]

JA98695: Chain of Custody

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DATA REVIEW WORKSHEETS

Project Number: JA98695
Date: 02/02/2012

REVIEW OF VOLATILE ORGANIC PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: USEPA Region 2, SOP HW-24, Standard Operating Procedure for the Validation of Organic Data Acquired using SW-846 Method 8260B (August, 2009-Revision 2), the USEPA National Functional Guidelines for Low/Medium Concentration Organic Data Review (SOW SOM01.2 SOP HW-33, August 2009 – Revision 2), the USEPA National Functional Guidelines for Organic Data Review for Low Concentration Water (SOP HW-13, August, 2009-Revision 3). Also, QC criteria from "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW-846 (Final Update III, December 1996)," specifically for Methods 8000/8015 are utilized. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Accutest data package received has been reviewed and the quality control and performance data summarized. The data review for VOCs included:

Lab. Project/SDG No.: JA98695 Sample matrix: SOIL
No. of Samples: 2

Trip blank No.: -
Field blank No.: -
Equipment blank No.: -
Field duplicate No.: -

<input checked="" type="checkbox"/> Data Completeness	<input checked="" type="checkbox"/> Laboratory Control Spikes
<input checked="" type="checkbox"/> Holding Times	<input checked="" type="checkbox"/> Field Duplicates
<input type="checkbox"/> GC/MS Tuning	<input checked="" type="checkbox"/> Calibrations
<input type="checkbox"/> Internal Standard Performance	<input checked="" type="checkbox"/> Compound Identifications
<input checked="" type="checkbox"/> Blanks	<input checked="" type="checkbox"/> Compound Quantitation
<input checked="" type="checkbox"/> Surrogate Recoveries	<input checked="" type="checkbox"/> Quantitation Limits
<input checked="" type="checkbox"/> Matrix Spike/Matrix Spike Duplicate	

Overall Comments: Isopropyl alcohol and methanol by SW-846_8015 (DAI)

Definition of Qualifiers:

J- Estimated results
U- Compound not detected
R- Rejected data
UJ- Estimated not detected

Reviewer: Rafael Infante
Date: 02/19/2012

DATA REVIEW WORKSHEETS

DATA COMPLETENESS

MISSING INFORMATION

DATE LAB. CONTACTED

DATE RECEIVED

[illegible]

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below

HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pH	ACTION
All samples were analyzed within the recommended method holding time.				

Criteria

Aqueous samples – 14 days from sample collection for preserved samples ($\text{pH} \leq 2$, 4°C), no air bubbles.

Aqueous samples – 7 days from sample collection for unpreserved samples, 4°C , no air bubbles.

Soil samples- 7 days from sample collection.

Cooler temperature (Criteria: $4 \pm 2^{\circ}\text{C}$) – 2°C

Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solid of soil samples is $< 10\%$, estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but < 14 days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but < 28 days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded (> 28 days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted ($> 10^{\circ}\text{C}$), estimate positive results (J) and nondetects (UJ).

DATA REVIEW WORKSHEETS

All criteria were met __N/A__
Criteria were not met see below _____

GC/MS TUNING

The assessment of the tuning results is to determine if the sample instrumentation is within the standard tuning QC limits

N/A The BFB performance results were reviewed and found to be within the specified criteria.

N/A BFB tuning was performed for every 12 hours of sample analysis.

If no, use professional judgment to determine whether the associated data should be accepted, qualified or rejected.

List the samples affected:

If mass calibration is in error, all associated data are rejected.

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

CALIBRATION VERIFICATION (VOCs)

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 01/12/2012
 Dates of continuing calibration: 02/07/2012 02/09/2012
 Instrument ID numbers: GCGH
 Matrix/Level: AQUEOUS/LOW

DATE	LAB FILE ID#	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
ALL INITIAL AND CONTINUING CALIBRATION MEET METHOD SPECIFIC CRITERIA				

Criteria

All RFs must be > 0.05 regardless of method requirements for SPCC.

All %RSD must be $\leq 15\%$ regardless of method requirements for CCC.

All %Ds must be $\leq 20\%$ regardless of method requirements for CCC.

It should be noted that Region 2 SOP HW-33 does not specify criterion for the curve correlation coefficient (r). A limit for r of ≥ 0.995 has therefore been utilized as professional judgment.

Actions

If any compound has an initial RF or a continuing RF of < 0.05 , estimate positive results (J) and reject nondetects (R), regardless of method requirements.

If any compound has a %RSD $> 15\%$, estimate positive results (J) and use professional judgment to qualify nondetects.

If any compound has a %RSD $> 90\%$, estimate positive results (J) and reject nondetects (R).

If any compound has a % D $> 20\%$, estimate positive results (J) and reject nondetects (R).

If any compound has a % D $> 20\%$, estimate positive results (J) and nondetects (UJ).

If any compound has a % D $> 90\%$, estimate positive results (J) and reject nondetects (R).

If any compound has r > 0.995 , estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/MATRIX	COMPOUND	CONCENTRATION UNITS
_____	_____	_____	_____	_____
All method blanks for this batch meet method specific criteria.				
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

Field/Equipment/Trip blank

DATE ANALYZED	LAB ID	LEVEL/MATRIX	COMPOUND	CONCENTRATION UNITS
No trip blank/equipment/field blank analyzed as part of this data package				
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

All criteria were met X
Criteria were not met
and/or see below

Blank Actions

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

ALs = 5x for any other compounds

Specific actions are as follows:

If the concentration is < sample quantitation limit (SQL) and \leq AL, report the compound as not detected (U) at the SQL.

If the concentration is \geq SQL but \leq AL, report the compound as not detected (U) at the reported concentration.

If the concentration is \geq SQL and $>$ AL, report the concentration unqualified.

Notes:

High and low level blanks must be treated separately

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

[illegible]

DATA REVIEW WORKSHEETS

All criteria were met _____
 Criteria were not met _____
 and/or see below X

SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

SAMPLE ID	SURROGATE COMPOUND				ACTION
	1,2-DCA	DBFM	TOL-d8	BFB	
JA94812-1				42%	No action taken
Surrogate Hexanol					

QC Limits* (Aqueous)

LL to UL to to to to

QC Limits* (Solid-Low)

LL to UL to to to 58 to 137

QC Limits* (Solid-Med)

LL to UL to to to to

1,2-DCA = 1,2-Dichloromethane-d4

TOL-d8 = Toluene-d8

DBFM = Dibromofluoromethane

BFB = Bromofluorobenzene

* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.

* If QC limits are not available, use limits of 80 – 120 % for aqueous and 70 – 130 % for solid samples.

Actions:

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	J
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

DATA REVIEW WORKSHEETS

All criteria were met _____
 Criteria were not met _____
 and/or see below X_____

VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID: JA98540-8MS/8MSD Matrix/Level: Soil/low
 Sample ID: JA98540-2MS/2MSD Matrix/Level: Soil/low

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
JA98540-2MS/-2MSD					
<u>MS</u>	<u>METHANOL</u>	<u>193</u>		<u>39-160</u>	<u>No action taken</u>
<u>MSD</u>		<u>195</u>			

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (JJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

All criteria were met X
Criteria were not met
and/or see below

MS/MSD – Unspiked Compounds

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID: N/A Matrix/Level/Unit: -

[illegible]

Actions:

- * If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).
* If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

1. LCS Recoveries Criteria

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD?
 Yes or No. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT

_____ Recoveries within laboratory control limits _____

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? **Yes** or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below

IX. LABORATORY/FIELD DUPLICATE PRECISION

Sample IDs: _____

Matrix: _____

Field duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: RPD \pm 30% for aqueous samples, RPD \pm 50 % for solid samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

[illegible]

Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

DATA REVIEW WORKSHEETS

All criteria were met N/A
Criteria were not met
and/or see below

X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- * Area of +100% or -50% of the IS area in the associated calibration standard.
- * Retention time (RT) within 30 seconds of the IS area in the associated calibration standard.

[illegible]

Actions:

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -25%	IS AREA = -25 % TO – 50%	IS AREA > + 100%
Positive results	J	J	J
Nondetected results	R	UJ	ACCEPT

2. If a IS retention time varies more than 30 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below

XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

JA98695-1 Hexanol (surrogate) RF = 61.69

$$[] = (130904)/(61.69)$$

$$[] = 2122 \text{ ppb OK}$$

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below _____

XII. QUANTITATION LIMITS

A. Dilution performed

[illegible]

B. Percent Solids

List samples which have $\leq 50\%$ solids

___ N/A ___

Actions:

If the % solids of a soil sample is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solids of a soil sample is < 10%, estimate positive results (J) and reject nondetects (R)

DATA REVIEW WORKSHEETS

Project Number: JA98695

Date: 02/02/2012

REVIEW OF VOLATILE ORGANIC PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: USEPA Region 2, SOP HW-24, Standard Operating Procedure for the Validation of Organic Data Acquired using SW-846 Method 8260B (August, 2009-Revision 2), the USEPA National Functional Guidelines for Low/Medium Concentration Organic Data Review (SOW SOM01.2 SOP HW-33, August 2009 – Revision 2), the USEPA National Functional Guidelines for Organic Data Review for Low Concentration Water (SOP HW-13, August, 2009-Revision 3). Also, QC criteria from "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW-846 (Final Update III, December 1996)," specifically for Methods 8000/8260B are utilized. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Accutest data package received has been reviewed and the quality control and performance data summarized. The data review for VOCs included:

Lab. Project/SDG No.: JA98695

Sample matrix: Soil

No. of Samples: 2

Trip blank No.: -

Field blank No.: -

Equipment blank No.: -

Field duplicate No.: -

☒ Data Completeness

☒ Holding Times

☒ GC/MS Tuning

☒ Internal Standard Performance

☒ Blanks

☒ Surrogate Recoveries

☒ Matrix Spike/Matrix Spike Duplicate

☒ Laboratory Control Spikes

☒ Field Duplicates

☒ Calibrations

☒ Compound Identifications

☒ Compound Quantitation

☒ Quantitation Limits

Overall Comments: Selected VOCs by SW846-8260B

Definition of Qualifiers:

J- Estimated results

U- Compound not detected

R- Rejected data

UJ- Estimated nondetect

Reviewer: Rafael Infante

Date: 02/19/2012

DATA REVIEW WORKSHEETS

DATA COMPLETENESS

MISSING INFORMATION

DATE LAB. CONTACTED

DATE RECEIVED

[illegible]

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below

HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pH	ACTION
All samples analyzed within the recommended method holding time				

Criteria

Aqueous samples – 14 days from sample collection for preserved samples ($\text{pH} \leq 2$, 4°C), no air bubbles.

Aqueous samples – 7 days from sample collection for unpreserved samples, 4°C , no air bubbles.

Soil samples- 7 days from sample collection.

Cooler temperature (Criteria: $4 \pm 2^{\circ}\text{C}$): 2°C - OK

Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimate positive results (J) and nondetects (UJ).

If the % solid of soil samples is $< 10\%$, estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but < 14 days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but < 28 days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded (> 28 days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted ($> 10^{\circ}\text{C}$), estimate positive results (J) and nondetects (UJ).

DATA REVIEW WORKSHEETS

If criteria were met ☒
Criteria were not met see below _____

Sample results qualified as estimated (J) in affected samples.

GC/MS TUNING

The assessment of the tuning results is to determine if the sample instrumentation is within the standard tuning QC limits

☒ The BFB performance results were reviewed and found to be within the specified criteria.

☒ BFB tuning was performed for every 12 hours of sample analysis.

If no, use professional judgment to determine whether the associated data should be accepted, qualified or rejected.

List _____ the _____ samples _____ affected:

If mass calibration is in error, all associated data are rejected.

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below _____

CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 01/12/12 01/18/12
 Dates of continuing calibration: 02/13/12 02/12/12
 Instrument ID numbers: GCMSV GCMSX
 Matrix/Level: Aqueous/low

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
Initial and continuing calibration meet method specific requirements					

Criteria

All RFs must be > 0.05 regardless of method requirements for SPCC.
 All %RSD must be $\leq 15\%$ regardless of method requirements for CCC.
 All %Ds must be $\leq 20\%$ regardless of method requirements for CCC.
 It should be noted that Region 2 SOP HW-24 does not specify criterion for the curve correlation coefficient (r). A limit for r of ≥ 0.995 has therefore been utilized as professional judgment.

Actions

If any compound has an initial RF or a continuing RF of < 0.05 , estimate positive results (J) and reject nondetects (R), regardless of method requirements.
 If any compound has a %RSD $> 15\%$, estimate positive results (J) and use professional judgment to qualify nondetects.
 If any compound has a %RSD $> 90\%$, estimate positive results (J) and reject nondetects (R).
 If any compound has a % D $> 20\%$, estimate positive results (J) and reject nondetects (R).
 If any compound has a % D $> 20\%$, estimate positive results (J) and nondetects (UJ).
 If any compound has a % D $> 90\%$, estimate positive results (J) and reject nondetects (R).
 If any compound has r > 0.995 , estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below _____

V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/MATRIX	COMPOUND	CONCENTRATION UNITS
		All_method_blank_meeth_method_specific_criteria		

Field/Equipment/Trip blank

[illegible]

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below _____

V B. BLANK ANALYSIS RESULTS (Section 3)

Blank Actions

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

ALs = 5x for any other compounds

Specific actions are as follows:

If the concentration is < sample quantitation limit (SQL) and \leq AL, report the compound as not detected (U) at the SQL.

If the concentration is \geq SQL but \leq AL, report the compound as not detected (U) at the reported concentration.

If the concentration is \geq SQL and > AL, report the concentration unqualified.

Notes:

High and low level blanks must be treated separately

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

CONTAMINATION SOURCE/LEVEL	COMPOUND	CONC/UNITS	AL/UNITS	SQL	AFFECTED SAMPLES

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

SAMPLE ID	SURROGATE COMPOUND				ACTION
	1,2-DCA	DBFM	TOL-d8	BFB	

 All surrogate recoveries within laboratory control limits

QC Limits* (Aqueous)

 LL to UL to to to to

QC Limits* (Solid-Low)

 LL to UL to to to to

QC Limits* (Solid-Med)

 LL to UL to to to to

1,2-DCA = 1,2-Dichloromethane-d4

TOL-d8 = Toluene-d8

DBFM = Dibromofluoromethane

BFB = Bromofluorobenzene

* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.

* If QC limits are not available, use limits of 80 – 120 % for aqueous and 70 – 130 % for solid samples.

Actions:

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	J
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

DATA REVIEW WORKSHEETS

All criteria were met _____
 Criteria were not met _____
 and/or see below X

VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID: JA98960-6 Matrix/Level: SOIL

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
<u>MS</u>	<u>ACETONE</u>	<u>190</u>		<u>12 - 189</u>	<u>No action; acetone not detected in sample JA98695-1</u>

Note: MS/MSD recoveries and RPD within laboratory control limits.

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

DATA REVIEW WORKSHEETS

All criteria were met _____
 Criteria were not met _____
 and/or see below X

VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID: JA98956-3 Matrix/Level: SOIL

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
<u>MS</u>	<u>ACETONE</u>	<u>236</u>		<u>12 - 189</u>	<u>Qualify_result_(J)</u>
					<u>in_sample</u>
					<u>JA98695-2</u>

Note: MS/MSD recoveries and RPD within laboratory control limits.

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (JJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below _____

VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

MS/MSD – Unspiked Compounds

It should be noted that Region 2 SOP HW-24 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID: _____ Matrix/Level/Unit: _____

COMPOUND	SAMPLE CONC.	MS CONC.	MSD CONC.	% RSD	ACTION
----------	-----------------	----------	-----------	-------	--------

[illegible]

Actions:

- * If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).
* If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

1. LCS Recoveries Criteria

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD?
 Yes or No. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT
<u>Recoveries within laboratory control limits</u>			

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

IX. LABORATORY DUPLICATE PRECISION

Sample IDs: JA98956-8

Matrix: SOIL

Laboratory duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: RPD \pm 30% for aqueous samples, RPD \pm 50 % for solid samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
RPD within laboratory and generally acceptable control limits					

Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below

X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- * Area of +100% or -50% of the IS area in the associated calibration standard.
- * Retention time (RT) within 30 seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE RANGE	ACTION
------	-----------	--------	---------	------------------	--------

Internal standard area within laboratory control limits

Actions:

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -25%	IS AREA = -25 % TO - 50%	IS AREA > + 100%
Positive results	J	J	J
Nondetected results	R	UJ	ACCEPT

2. If a IS retention time varies more than 30 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below

XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

JA98695-2

Acetone RF = 0.027

$$[] = (5389)(50)/(358489)(0.027)$$

$$= 27.8 \text{ ppb OK}$$

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below _____

XII. QUANTITATION LIMITS

A. Dilution performed

[illegible]

B. Percent Solids

List samples which have $\leq 50\%$ solids

Actions:

If the % solids of a soil sample is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solids of a soil sample is $< 10\%$, estimate positive results (J) and reject nondetects (R)